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(71) Applicants
David Roberts,
39 Stumperlowe Crescent Road, Sheffield S10 3PR
Henry Stuart Johnson,
6 Slayleigh Lane, Sheffield S10 3RF

(72) Inventors
David Roberts,
Henry Stuart Johnson

(74) Agent and/or Address for Service
Hughes Clark Byrne & Parker, 63 Lincoln's Inn Fields,
London WC2A 3JU

(54) Vitrification of inorganic materials

(57) In the conversion of a toxic inorganic material into a glass an aqueous solution or sludge of the inorganic material is agitated with anhydrous sodium carbonate or other anhydrous material in super-stoichiometric amount to form a dry flowable powder that is melted with other glass-making materials.

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SPECIFICATION

Vitrification of inorganic materials

This invention relates to a process for converting aqueous solutions of toxic inorganic materials into non-toxic glass for safe disposal.

The chemical industry produces aqueous sludges of silica and alumina based catalysts containing heavy metals. These toxic sludges present a safe disposal problem. A typical such catalyst sludge contains 50-60% water, the balance being organic residues and spent catalyst. The inorganic materials present can in principle be converted into a glass that is stable against heavy metal leaching, and the resultant glass can then be disposed of in a safe location that may even be a landfill site. But the conversion of these sludges into glass presents the problems that (a) the sludge needs to be converted into a form in which it is compatible with other glass-making materials, and (b) the comparatively high concentrations of organic material and water present can present problems in the control of the glass chemistry.

We have found that spent catalyst sludges can be converted by the addition of anhydrous sodium carbonate into a dry handleable powder which can be further converted by melting with glass-making materials into a homogeneous glass such as a soda-lime glass. Other anhydrous materials that chemically combine with water and may be used include sodium sulphate, borax, calcium oxide, a sodium silicate such as sodium metasilicate and magnesium sulphate. Materials that physically absorb water may also be used.

Broadly stated the invention provides a method of converting a toxic inorganic material into a glass, comprising agitating an aqueous solution or sludge of the inorganic material with an anhydrous material that physically or chemically absorbs water and is compatible with glass making in a super-stoichiometric amount so that the sludge is converted into a dry flowable powder; and melting the powder with glass-making materials to form a glass.

Sodium carbonate is the preferred anhydrous material and the amount to be added will typically be 50-100 parts per 100 parts by weight of the sludge. The sludge and sodium carbonate are simply stirred together at a temperature below 32 degrees C to convert the sodium carbonate into its decahydrate. Addition of sodium carbonate in amounts above the stoichiometric amount results in conversion of the sludge into a dry free-flowing powder.

Glass may be made from the powder in amounts of 10-40% of the total weight of glass-making material, but it may be desirable to agitate the material e.g. by bubbling in order to produce a homogeneous material.

The invention will now be described with reference to the following examples:

Example 1

A sludge containing water, organic residues and a catalyst containing heavy metals and silica was treated with sodium carbonate in the amounts and with the results indicated in Table 1 below:

TABLE 1

Sodium carbonate drying trials

Test No.	*Sludge Wt*	*Sodium Carbonate Wt*	*Remarks*
1	100	40	Formed powder but there is a residual stickiness.
2	100	60	Free-flowing dry powder.
3	100	80	As 2.

The sludge of Test No.3 was used to make 40g pot melts as indicated in Table 2 and with th results there quoted. Although inhomogeneity was observed in many of the melts, this is believed to have been due to the absence of stirring, and it is believed that this inhomogeneity would not be observed using a larger scale melter with an air bubbling system to stir the glass and oxidise materials present. The analyses of the glasses produced are as shown in Table 3.

TABLE 2

Melting trials

Table No.	Sludge	Sludge/ Soda (a)	Na_2CO_3	Sand	Limestone	$NaNO_3$	Na_2SO_4	Bottle Cullet	Test 2-8 Glass	Soda lime batch	Remarks
1	100	-	37	-	-	-	-	-	-	-	Viscous glass much undesolved material
2	-	50.7	-	67.5	17.7	0.74	-	-	-	-	)
3	-	50.7	-	67.5	17.7	0.925	-	-	-		) Mainly glass but significant streakiness - no significant difference with changes in nitrate
4	-	50.7	-	67.5	17.7	1.11	-	-	-	-	)
5	-	50.7	-	67.5	17.7	3.5	-	-	-	-	)
6	-	50.7	-	67.5	17.7	-	2.7	-	-	-	Not markedly better than 2 - 5
7	-	50.7	-	67.5	17.7	-	8.1	-	-	-	Noticeably better but still streaking
8	-	50.7	-	67.5	17.7	3.5	8.1	-	-	-	As 7
9	-	-	-	-	-	-	-	50	50	-	Deep amber glass with not streaks
10	-	28.0	-	37.3	9.8	-	-	-	-	25	Good glass but streaky, as 7
11	-	26.1	-	34.8	9.1	-	-	-	-	25	As 7
12	-	26.1	-	34.8	9.1	-	1.4	-	-	25	As 7
13	-	18.7	-	24.8	6.5	-	-	-	-	50	As 7
14	-	6.0	-	8.0	2.1	-	-	-	-	84	Blue coloured glass with amber streaks

(a) The composition of this was 100 g sludge, 40 g sodium carbonate.

TABLE 3

Glass composition
Oxides % W/W

Melt Number	SiO_2	Na_2O	CaO	Sb_2O_3	UO_2	MoO	Al_2O_3
1	28.0	54.5	-	15.4	1.7	0.5	-
2	76.8	9.4	11.1	2.4	0.26	0.08	-
3	76.7	9.5	11.1	2.4	0.26	0.08	-
4	76.7	9.5	11.1	2.4	0.26	0.08	-
5	76.0	10.4	11.0	2.3	0.25	0.08	-
6	76.0	10.3	11.0	2.3	0.25	0.08	-
7	74.0	12.7	10.7	2.3	0.25	0.08	-
8	73.0	13.8	10.5	2.3	0.25	0.08	-
9	73.2	13.7	10.5	1.15	0.13	0.04	0.5
10	75.7	10.5	11.0	1.7	0.18	0.06	0.2
11	75.6	10.6	11.0	1.7	0.18	0.06	0.2
12	75.4	11.4	10.9	1.6	0.17	0.06	0.2
13	75.5	11.6	11.1	1.07	0.15	0.03	0.6
14	74.5	13.1	11.2	0.32	0.04	0.01	0.87

Example 2

A spent catalyst sludge or so-called "pond sludge" having the composition given in Table 4 was conditioned by adding anhydrous sodium carbonate to the sludge, whilst stirring, at a temperature below 32°C. The formation of sodium carbonate decahydrate absorbed all the water - and by increasing the amount of carbonate, sufficient of the organic liquids as well. The resultant dry powder was used a sodium source to produce a conventional soda-lime glass. The test was intended to demonstrate that the sludge could be totally absorbed into a finished glass and that the high level of organic materials did not interfere with glass making. After early attempts where some of the metals were not absorbed, a mix and melting schedule were found which gave a homogeneous black glass.

TABLE 4

Pond sludge composition

Uranium	% w/w	0.59
Molybdenum	% w/w	0.18
Antimony	% w/w	5.1
Free Cyanide	ppm	0.2
Total Cyanide	% w/w	0.038
AMM N_2	% w/w	0.97
Ash	% w/w	17.0
Water	% w/w	63.0
Orgs + AMM Sulphate	%	20.0

CLAIMS

1. A method of converting a toxic inorganic material into a glass, comprising:
agitating an aqueous solution or sludge of the inorganic material with an anhydrous material that physically or chemically absorbs water and is compatible with glass making in a super-stoichiometric amount so that the sludge is converted into a dry flowable powder; and
melting the powder with glass-making materials to form a glass.

2. A process according to claim 1, wherein the anhydrous material is sodium sulphate, borax, calcium oxide, sodium silicate or magnesium sulphate or a xeolite.

3. A process according to claim 1, wherein the anhydrous material is sodium carbonate.

4. A process according to claim 3, wh rein the anhydrous sodium carbonate is added in an amount of 50-100 parts by weight per 100 parts by weight of solution or sludge.

5. A process according to claim 3 or 4, wherein the solution or sludge is maintained at a temperature below 32 degrees C during conversion to the dry powder.

6. A process according to any preceding claim wherein the dry powder is added in an amount of 10-40% by weight based on the total weight of the glass-making materials.

7. A process according to any preceding claim, wherein the glass-making materials include sand and limestone.

8. A process according to any preceding claim, wherein the catalyst is a heavy metal supported on silica or alumina and is present in the sludge together with organic materials.

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